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ENVIRONMENTAL REACTIONS OF ROCKET STEELS

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ENVIRONMENTAL REACTIONS

OF

ROCKET STEELS

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INTRODUCTION

Unique and interesting engineering problems have arisen from the use of very high strength materials in rocket construction. For example, it is a general rule of thumb that increasing strength is usually accompanied by increasing susceptibility to embrittlement and stress corrosion phenomena. Since rocket cases and components are exposed to a wide variety of potentially corrosive environments in fabrication, testing, storage, service, etc.,an evaluation of the influence of environmental factors upon rocket materials is of interest. In this program the fracture behavior of high strength sheet steels in typical aggressive environments is under investigation. In these preliminary stages the research has been directed to the following goals:

1) To determine quickly the important parameters controlling stress corrosion in aqueous environments.

2) To adapt the electric resistance method of following crack initiation and propagation to the present investigation. This method should allow a detailed study of the events preceeding crack instability and fracture in both the presence and absence of corrosive environments.

MATERIALS AND PROCEDURE

The following steels are under study:

(1) AMS 355, 3 in. x .078 in., heat treated to a yield strength of 210,000 psi., notched by Elox machining to a root radius of approximately .001 in.

(2) X-200; 3 in. x .068 in., heat treated to a yield strength of 230,000 psi., notched by Elox machining to a root radius of approximately .001 in.

Mechanical testing is performed in an Amsler machine with grips prepared from NRL drawings. For corrosion exposure tests a small plastic box is clamped

on the specimen center section. The box is then filled with distilled water or with gaseous environments such as argon and argon saturated with water vapor. The environmental influence is evaluated currently by determining the number of three minute load cycles to failure, and comparing this with the performance of an unexposed specimen.

The electric resistance method of following crack growth utilizes a Kelvin Double Bridge for precision measurements of low resistances. With initiation and growth of a crack, the cross sectional area of the specimen decreases; this results in an easily observed increase in electric resistance. Previous investigations have shown this technique to be quite accurate.

RESULTS AND DISCUSSION

In the initial stages of the project, while the electric resistance apparatus was assembled, a number of mechanical tests were performed to indicate the magnitude of the stress corrosion effect. These tests will be discussed first, and will be followed by preliminary results with the resistance method. Tables I and II summarize the mechanical test results to date in terms of the net section fracture stress and the number of cycles to failure.

It is apparent that the two steels differ significantly both in inherent fracture toughness and in response to the corrosive environment. The net section fracture stress values show that the AMS 355 steel is inherently tougher than the X-200 steel in the absence of a corrosive environment. This perhaps reflects differences in both chemical composition and strength level.

Further, the corrosive environments (water or argon saturated with water vapor) drastically affect the fracture behavior of the X-200 steel, yet have little or no influence upon the AMS 355. For comparable net section stresses the water-exposed AMS 355 consistently fractures in fewer cycles than the

unexposed steel; however, the differences are small and probably would not be significant in terms of rocket motor case performance.

It is of interest to note (Tests 3-2 and 3-3) that prolonged cycling at stresses well in excess of one-half of the yield strength does not influence the fracture behavior of AMS 355 upon subsequent stressing. This appears to be true for both water exposed and unexposed specimens.

With X-200 steel the influence of the corrosive environment is so severe as to indicate that normal fracture tests may be influenced by environmental factors. Water and water vapor saturated argon appear to be of comparable severity; each will produce fracture at a net section stress of 28,500 psi. in a material whose nominal yield strength is 230,000 psi. These observations, when considered in conjunction with the lack of influence of the corrosive environment on the AMS 355 steel, suggest strongly that the effect of strength level upon the fracture behavior of a single material should be determined.

A point of fundamental interest is the influence of test temperature upon fracture behavior in the presence of a corrosive environment. A single test (1-19) with an H_2O-ice mixture indicated a larger number of cycles to failure than observed with room temperature water. However, the difference may not exceed normal scatter and, in any event, the experimental technique needs refinement. This approach will be explored further, since insight into the corrosion fracture mechanism may well result.

Preliminary results substantiate the thought that the electric resistance method is well suited to detecting the presence or absence of slow crack propagation under the experimental conditions of this investigation. The controlled growth of a crack in the presence of distilled water is presented in Table 3. An X-200 specimen was subjected to eleven three minute cycles at 36,500 psi., followed by four cycles at 43,000 psi. The specimen was then

removed unbroken from the machine and radiographed. During the first eleven cycles the crack growth rate was very slow and irregular, and there was no apparent incubation period for crack initiation. The increase in net section stress to 43,000 psi. was soon accompanied by a striking increase in crack propagation rate, and the growing crack was easily visible. The crack lengths measured on the specimen surface and on the radiograph were in agreement. Further, metallographic examination of the crack at successive depths indicated that the crack length in this particular specimen was constant from the specimen surface to the mid-thickness point. This seems to be a rather important observation, and it will be further investigated.

The influence of water vapor saturated argon upon fracture behavior is vividly illustrated in Table 4 for X-200 stressed at 28,500 psi. Twice during the test the corrosive environment was removed and then reapplied. In both instances a decrease in crack growth rate was evident; after the second removal, the crack virtually halted in cycles 25 through 28. When the corrosive environment was reinstated, rapid crack growth and fracture ensued. A continual supply of corrosive environment is apparently necessary for continued crack growth; once again, further experimentation is desirable.

Slow crack propagation has not yet been observed in X-200 tested in the absence of a corrosive environment. Table 5 confirms this for X-200 tested at a maximum cycle stress of 79,000 psi. Nine three minute cycles were endured without crack formation; the specimen fractured immediately upon reaching maximum stress in the tenth cycle. Since slow crack propagation is normally observed in X-200, the present result suggests that the crack starting defect was of insufficient sharpness. These experiments will be further pursued with specimens containing notches of maximum possible sharpness.

The increase in resistance with increasing stress during a normal test is shown in Table 6. At low stresses this resistance increase is attributed primarily to elastic deformation, since it disappears upon unloading. If either substantial plastic flow or crack formation occurs, the resistance increase will not disappear. In this case there were no resistance increases which could be attributed to crack formation. The entire fracture process was nearly instantaneous.

SUMMARY AND CONCLUSIONS

These preliminary results show clearly that very high strength steels may be susceptible to failure at unexpectedly low stresses in the presence of an aggresive environment. However, the factors controlling the magnitude of the stress corrosion effect are not yet well defined, although both chemical compositon and strength level seem to play important roles. Thus, the AMS 355 steel is virtually unaffected by aqueous environments, while the X-200 at a slightly higher strength level shows substantial effects of stress corrosion.

The potential usefulness of the electric resistance method in experimental fracture mechanics is shown by the results on slow crack propagation in X-200 steel. The utility of the method will be further enhanced when a calibration is available to convert resistance increases into crack propagation rates. This is to be approached from both a theoretical and experimental point of view.

Table I. Cyclic Load Fracture Behavior of AMS 355 Steel

Test No.	Environment	Net Section Stress	No. 3 min. Cycles to Failure	Observation
3-4	none	173,500 psi.	on loading	nearly completely
3-5	water	181,000	on loading	shear fracture
3-17	argon sat. with H_2O	176,000	on loading	
3-6	none	169,000	4.05	" "
3-7	water	169,000	1	" "
3-8	none	160,000	7	" "
3-9	water	160,000	6.33	" "
3-10	none	155,000	17.15	" "
3-11	water	155,000	14.85	" "
3-12	none	148,000	> 25	some crack growth
3-13	water	148,000	> 25	visually evident

3-2 no water (1) 10 cycles at net section stress = 123,500 psi.
(2) 8 cycles at net section stress = 154,000 psi.
(3) Upon further loading fractures at 170,000 psi.

3-3 water (1) 10 cycles at net section stress = 123,500 psi.
(2) 9 cycles at net section stress = 154,000 psi.
(3) Upon further loading fractured at 170,000 psi.

Table 2. Cyclic Load Fracture Behavior of X-200 Steel

Test No.	Environment	Net Section Stress	No. 3 min. cycles to failure	Observation
1-1	none	110,000 psi.	on loading	substantial brittleness in all fracture surfaces
1-2	water	71,750	"	
1-3	none	95,000	2.9	
1-5	none	85,000	12.85	
1-15	none	85,000	6	
1-14	argon	85,000	3	
1-17	argon sat. with H_2O	85 000	on loading	cracking prior to fracture
1-18	argon sat. with H_2O	85,000	0.4	
1-7	none	79,000	15.8	
1-12	none	71,500	$>$ 25	
1-4	water	64,000	0.4	
1-6	water	57,000	0.05	
1-8	water	50,000	0.05	
1-9	water	43,000	4	
1-16	water	43,000	8.4	
1-19	ice-E_2O mixture	43,000	10.4	
1-11	water	36,500	4.4	
1-13	water	28,500	$\geq$25	substantial crack growth visually observed
1-21	argon sat. with H_2O	64,000	0.17	
1-22	" " " "	57,000	0.35	
1-23	" " " "	50,000	3.0	
1-24	" " " "	43,000	0.67	
1-25	" " " "	43,000	1.17	
1-30	" " " "	28,500	32.0	

Table 3. Slow Crack Propagation in X-200 Subjected to Cyclic Stressing in a Distilled Water Environment

Cycle	Max. Stress	Resistance Increase During Time at Max. Stress
1	36,500 psi.	16×10^{-8} ohm
2	"	20 "
3	"	12 "
4	"	0 "
5	"	16 "
6	"	16 "
7	"	4 "
8	"	20 "
9	"	4 "
10	"	12 "
11	"	12 "
12	43,000	12 "
13	"	152 "
14	"	260 "
15	"	2480 "

Specimen then removed from machine.

Table 4. Slow Crack Propagation X-200 Subjected to an Argon-Water Vapor Environment. Stresses at 28,500 psi.

Cycle	Resistance Increase
1	56 x 10^{-8} ohm
2	40
3	40
Corrosive environment removed	
4	4
5	16
Corrosive environment returned	
6	12
7	12
8	16
9	28
10	8
11	32
12	12
13	20
14	28
15	24
16	32
17	52

Cycle	Resistance Increase
18	44
19	84
20	100
corrosive environment removed	
21	36
22	60
23	44
24	64
25	4
26	12
27	8
28	8
Corrosive environment returned	
29	40
30	168
31	316
32	996
33	>5600

Table 5. Lack of Slow Crack Propgation in X-200 Tested in the Absence of Corrosive Environment. Net Section Stress 79,000 psi.

Cycle	Resistance Increase
1	16×10^{-8} ohm
2	-8
3	-4
4	0
5	8
6	-4
7	-4
8	0
9	0

Specimen fractured upon attaining maximum stress in 10th cycle. No visual evidence of slow crack growth prior to fracture.

Table 6. Resistance Increase During Normal Test of X-200 Steel

Net Section Stress	Resistance Increase
0	0
14,200 psi	32×10^{-8} ohm
28,500	96
43,000	128
57,000	140
71,000	212
85,000	248
96,000	264

Fracture at 99,000 psi.